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Study of structural evolution of chars during rapid pyrolysis of waste CDs at different temperatures



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HIGHLIGHTS

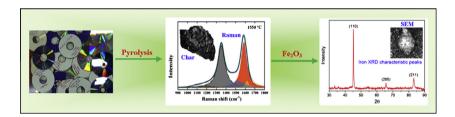
- Rapid pyrolysis was performed for waste CDs at different temperatures (550–1550 °C).
- Various analytical techniques were used to study the structure of evolved chars
- At higher pyrolysis temperature, the evolved chars were highly ordered and aromatic.
- The obtained char was used as reductant in ironmaking industries.
- Waste CDs char indicates as a valuable supplementary carbon resource for ironmaking.

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ABSTRACT

In this paper, the work dealt with the characterisation of chars obtained by rapid pyrolysis of waste compact discs (CDs) at different temperatures and its application as reductant in ironmaking industries. The rapid pyrolysis was performed at atmospheric pressure and temperatures ranging from 550 to 1550 °C. The obtained chars were characterised by ultimate analysis, X-ray diffraction (XRD), Raman spectroscopy, Fourier transform infrared spectroscopy (FTIR), X-ray photon spectroscopy (XPS), N2 isothermal adsorption method and scanning electron microscopy (SEM). The results indicated that char structure changed upon different pyrolysis temperature. The char yield decreased from 22% to 18% and gradual increase in carbon/oxygen ratio was observed with increase in pyrolysis temperature from 550 to 1550 °C. The porosity in chars increased progressively with pyrolysis temperature, and maximum development of pores appeared at 850 °C with surface area 334 m²/g. At higher pyrolysis temperature, the chars became more aromatic and ordered, and aliphatic structures decreased significantly. The loss of functional groups at higher temperature such as carbonyl, aliphatic C—H was evident by FTIR, XPS and Raman results. The ordered char structure obtained at 1550 °C was used as reductant carbon for iron oxide reduction process, and 90% reduction was achieved. The waste CD char obtained during pyrolysis indicates as a valuable supplementary carbon source for ironmaking industries.

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1. Introduction

In the developed world, 75% of the population live in urban areas, it is expected to grow 85% by 2030, while in the developing

world, the rate of urbanisation is even faster [1,2]. Due to rapid urbanisation and rise in standards of living, the amount of waste generated has significantly increased during last decade [3]. Electronic waste is considered as one of the rapidly emergent problem, increasing three times faster than normal municipal waste at the rate of 3–5% per annum all over the world [4]. Among electronic wastes, compact discs (CDs) and digital versatile discs (DVDs) are

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devices which are widely used for storing text, music, computer programs, software's and videos. CDs and DVDs are used extensively among any other storage devices such as video tapes, cassettes, and floppy disc. According to the United States Environmental Protection Agency, nearly 5.5 million CDs go to landfills and incinerators every year, and millions of CDs are just thrown away [5]. The nature of CDs does not allow it to recycle easily due to its heterogeneous combination of polymer and multi-layer metal coating. Among physical, chemical and pyrolytic techniques to recycle waste CDs [6-8], pyrolytic has been recognized as promising process. Rapid pyrolysis at high temperature plays an important role as it is first step in gasification process. The pyrolysis conditions determine the char yield and structure. The char structure evolved during different pyrolytic conditions is very beneficial for various applications [9,10]. The waste CD pyrolysis yields gases and significant percentage of solid residue [11]. The typical char residue contains carbon, which can be used as fuel or chemical feedstock. In this aspect, it is very significant to study the char structure evolved at different temperatures during rapid pyrolysis of waste CDs.

The iron and steelmaking industries are one of the major consumers of coal/coke in the world. Coal and coke are used as carbon sources for iron oxide reduction, alloy making, as carburiser and also for synthesis of refractory materials. Extensive research has being carried out on using the waste materials as supplementary carbon source to reduce the consumption of coal/coke in steelmaking industries. The use of agricultural and rubber waste has received significant attention due to the presence of high amount of carbon [12,13]. The use of wood char in ironmaking has been extensively reviewed by Kumar and Gupta [14]. Charcoal has been used as a reducing agent in ironmaking for number of years. Research from our group has shown the use of waste plastic and rubber tyres as partial replacement of coke in electric arc furnace (EAF) steelmaking [15,16]. Recently, our research focused on gas evolution studies and decomposition mechanism of waste CDs during pyrolysis [11]. The aim of the present work is to characterise the chars obtained by rapid pyrolysis of waste CDs at different temperatures and application of obtained char as supplementary carbon source in ironmaking industries.

In this research, the focus is to understand the yield and structural evolution of chars obtained by waste CDs at different rapid pyrolytic temperatures. The rapid pyrolysis experiments were performed at atmospheric pressure and at different temperatures ranging from 550 to 1550 °C for 20 min. The characterisation was done by ultimate analysis, X-ray diffraction (XRD), Raman spectroscopy, Fourier transform infrared spectroscopy (FTIR), X-ray photon spectroscopy (XPS), N_2 isothermal adsorption method and scanning electron microscopy (SEM). The obtained char was efficiently used as reductant carbon for iron oxide reduction process.

2. Experimental

2.1. Material

The material used is a typical waste CDs. Prior to experiments, the sample was first crushed and pulverized, and was dried in an oven at 105 °C for 3 h. The ultimate and elemental analyses were performed to determine the elements present in waste CDs. The proximate analysis was also performed to determine the moisture, volatile and fixed carbon in waste CDs. Results of ultimate, proximate and elemental analysis are shown in Table 1.

2.2. Pyrolytic procedure

The rapid pyrolysis of waste CDs was carried out under atmospheric pressure in a horizontal tubular furnace in an argon atmosphere. The hot zone of the furnace was preheated to the desired temperature (550–1550 °C) and it was controlled by a thermocouple. A weighed amount of waste CD sample was kept in a crucible and placed on a sample holder. Initially the sample holder was placed at the cold zone to avoid thermal shock, held there for 5 min, and then pushed into the hot zone. After 20 min, the samples were withdrawn from the furnace, and then cooled down in argon atmosphere to room temperature. The chars were collected and weighted to determine the yield. The pyrolysis gases were allowed to escape the furnace and were not recovered; only solid chars were analysed.

2.3. Characterisation methods

Ultimate analysis was performed to determine the percentage of carbon and oxygen in chars obtained at different temperatures, which is usually reported as the wt% of the respective element.

The crystalline components of chars were analysed by PANalytical X'Pert Pro multipurpose X-ray diffractometer, operating at 40 kV and 40 mA and measurements were recorded from a start angle $2\theta = 10^{\circ}$ to an end angle of 80° , with total accumulation time of around 30 min.

Raman spectra were also acquired for chars to determine the crystallinity and molecular structure using Renishaw Invia Raman Microscope. Spectra were measured at constant room temperature using 514 nm wavelength. The laser was focused through a microscope with a 50X objective. Each Raman spectra presented in this study corresponds to accumulation of 4 spectra's recorded from 800 to 1800 cm⁻¹ over 30 s and, curve fitted software Magic plot was used to determine respective Lorentzian bands.

FTIR analysis was performed to study the surface chemistry of chars. The chars were powered and mixed with KBr in a mass ratio of 1:100, and then the samples were scanned using Spectrum 100, PerkinElmer Fourier transform infrared spectroscopy in wavenumber range of 4000–650 cm⁻¹ to analyse the functional groups in chars.

XPS analysis was carried out to study the chemical state of carbon in chars obtained during temperature 550 °C and 1550 °C at rapid pyrolysis. The Al K α line was used as the X-ray source. The

Table 1Ultimate, proximate and elemental analysis of waste CDs.

Proximate analysis (wt% as received) Moisture	0.22
Ash	2.02
Volatile matter	77.99
Fixed carbon	19.77
Ultimate analysis (wt% as received)	
C	76.03
0	20.62
N	00.21
Elemental analysis (X-ray fluorescence studies)	
Analyte	Concentration (%)
F	0.914
Na	0.036
Mg	0.044
Al	0.070
Si	2.160
P	0.007
S	0.028
Cl	0.074
Ca	0.750
Fe	0.058
Ni	0.004
Br	0.005
Ag	0.142
Bi	0.004

photoelectron signals were detected from the sample surface at the 90° angle, at which the detected sensitivity is enhanced. For measurements, samples were loaded into ultra-high vacuum (about 2×10^{-9} mbar) chamber of the XPS apparatus.

The physical structure of chars was described using SEM. A coating of gold film was applied to samples using a Cressington 208 HR Sputter Coater. The coated samples were then examined and imaged using a Topcon SM720 Field Emission Scanning Electron Microscope.

The surface area of chars was studied by means of N_2 physisorption on a Micrometrics Tristar absorption analyser. Prior to analysis, all the samples were dried at 110 °C in oven and then degassed for at least 3 h at 150 °C under vacuum. The total specific area, $S_{\rm total}$ (m²/g) of the samples is evaluated using the multilayer adsorption model developed by Brunauer, Emmett and Teller.

2.4. Reduction of iron oxide procedure

Waste CD char obtained at 1550 °C for 20 min by rapid pyrolysis was used for reduction of iron oxide studies. The char was grounded to an upper size limit of 125 μm using a ring mill. In reduction experiment, iron oxide used was haematite (99% pure) with a particle diameter of 5 μm . The iron oxide and char were thoroughly mixed using rolling mill for 4 h. The mole ratio of carbon to oxygen in the mixture was $\sim\!1.2$. The reduction experiments were performed in horizontal tubular furnace. The reduction studies were carried out at different temperatures (1200 °C and 1400 °C) in an argon atmosphere at flow rate of 1.0 L/min for 20 min residence time.

Off gas analysis during the reduction of iron oxide was carried out using IR analyser (ABB, Advance Optima Series AO2020) to record the amount of CO_2 and CO formation. The gas concentrations of CO and CO_2 were used to determine the cumulative amounts of gases coming during reduction process. The reduced mixtures were analysed by XRD and SEM.

3. Result and discussion

3.1. Char yield and ultimate analysis

The term 'char' is used represent the solid residue obtained after pyrolysis. Thermogravimetric analysis (TGA) was performed to gain knowledge of pyrolysis of waste CDs. Fig. 1 exhibits the TG curve for pyrolysis of waste CD at a heating rate of 10 °C/min in nitrogen atmosphere from room temperature to 1550 °C. The degradation of waste CD started around 550 °C was clearly observed from TGA curve, and hence the rapid pyrolysis studies were carried out from 550 °C to 1550 °C. Fig. 2 shows the effect of temperature on char yield during rapid pyrolysis. It can be observed that the char yield decreases from 26% to 19% when the pyrolysis temperature increased from 550 to 1550 °C. The decrease in the yield is due to increase in primary or secondary decomposition in char residue at higher temperatures. The progressive increase in carbon content and decrease in oxygen content with pyrolysis temperature was observed in chars. The increase in carbon/oxygen ratio (Fig. 2); clearly indicates the increase of aromatic and carbonaceous nature in chars. The chemical analysis of char obtained at 1300 °C shows around 90% of carbon (Table 2).

3.2. Characterisation of chars

3.2.1. XRD analysis

The structural ordering of char structure as the function of pyrolysis temperature was investigated by XRD technique. Fig. 3 presents the XRD profiles of waste CD chars produced at different

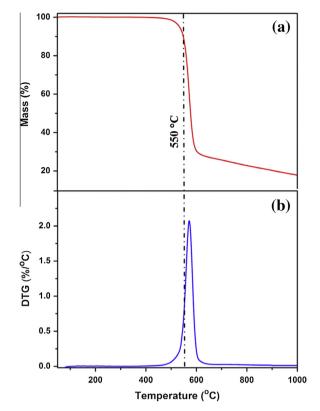


Fig. 1. (a) TGA and (b) DTG curve of waste CD.

pyrolysis temperature. Note that all spectra's were arranged vertically on the same scale of 2θ in order to show the shift of peaks. In all spectra's, two peaks were observed clearly over the examined range (10-80°), corresponding to the diffuse graphite (002) and (100) peaks in low and high 2θ regions, respectively [17]. The (002) peak can be attributed due to the consequences of a parallel packing of carbon layers, with a distance between layers in a stack. Theoretically, (002) peak should be symmetric, but exhibits asymmetric nature mostly for low scattering angles because of presence of γ -band. It is assumed that γ -band is associated with the amorphous structure, presence of ordering in aliphatic structure, or because of irregularity in aromatic structures packing. As the pyrolysis temperature increases, the background intensity of the spectra for waste CD chars became weaker and (002) peak became more symmetric and sharper. The shift of 2θ angle as the function of temperature towards higher angle shows the decrease in contents of amorphous carbon and aliphatic structure in the char and increase in ordering in aromatic structure [18]. The (100) peak is interpreted as a result of two dimensional reflection of X-ray from carbon layers, which also characterises the aromatic part of the char. The (100) peak at $44-45^{\circ}$ is considered by aromatic ones and confirms the presence of aromatic nature carbon in chars. However, the change in (100) peaks is less significant in magnitude than compared with (002) peaks. Therefore, the disorder in parallel orientation is expected to be more in lower temperature chars. The inter-planar spacing of two crystal lattices in chars decreased from 0.5 nm at 550 °C to 3.1 nm at 1550 °C, indicating increase in crystallinity as pyrolysis temperature increases.

3.2.2. Raman analysis

Raman spectroscopy is a powerful technique as it is sensitive to both molecular and crystalline structure, and widely employed to characterise carbonaceous materials. In this study, Raman spectroscopy is used to understand the effect of temperature on

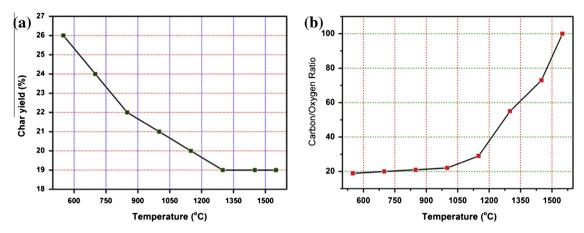


Fig. 2. Change of (a) char yield and (b) carbon/oxygen ratio during rapid pyrolysis of waste CDs at different temperatures.

Table 2 Elemental analysis of chars obtained at 550 °C and 1550 °C temperatures.

Major elements	Char 550 °C	Char 1550 °C		
Elemental analysis of chars (concentration, %)				
C	86.21	89.82		
0	4.505	0.904		
Si	6.121	6.662		
Ca	2.130	2.385		
F	0.585	0.030		
Ag	0.125	0.004		
Others (Na, Mg, Al, Fe)	0.324	0.095		

crystallinity and molecular structure of char obtained during rapid pyrolysis of waste CDs. All spectra's of char showed 2 dominant peaks i.e. G band (\approx 1590 cm⁻¹) and D band (\approx 1350 cm⁻¹). For hexagonal graphite, 'G band' is assigned to $E_{\rm 2g}$ vibrational mode, which is attributed to in plane stretching motion pairs of carbon sp² atoms. Another band originates due to existence of disorder in graphitic structure which is assigned to A_{1g} symmetry and is named as 'D band'. Furthermore a peak related to random structure, hides under valley between two peaks, which reflects the presence of amorphous carbon (Fig. 4a). In this study, the I_V/I_G ratio (I_V is intensity of a valley between G and D bands and I_G is intensity of G band) is been used as an indication to measure the degree of disorderness and proportion of amorphous carbon. From Fig. 4b, it was clearly observed that I_V/I_G ratio progressively decreased in chars with higher pyrolysis temperature. The I_V/I_G ratio decreased from 0.60 to 0.33 in chars when the pyrolysis temperature was increased from 550 °C to 1550 °C. A decrease in I_V/I_G ratio indicates the decrease of amorphous contents, consequently reducing the disordered contents and increasing the crystallinity in chars.

In order to understand the molecular structures in chars obtained at 550 °C and 1550 °C, the deconvolution of spectra's was performed (Fig. 5). Deconvolution of spectra's induced additional three bands with D band designated as D2 (1620 cm⁻¹), D3 (1530 cm⁻¹) and D4 (1150 cm⁻¹). The intensities of these bands directly relates to disorderness, amorphous content and oxygen containing functional groups in char. The D2 band exists due to presence of C—H on aromatic groups and C—C on hydroaromatic groups, D3 band is result of 3–5 rings aromatic structures with amorphous contents, and D4 band relates to the oxygen containing functional (C—O and C=O) groups. The decrease in intensity of D2, D3 and D4 bands in 1550 °C char compare to 550 °C char indicates the reduction of functional groups and amorphous content thus indicating an increase of structural ordering in 1550 °C char [19,20].

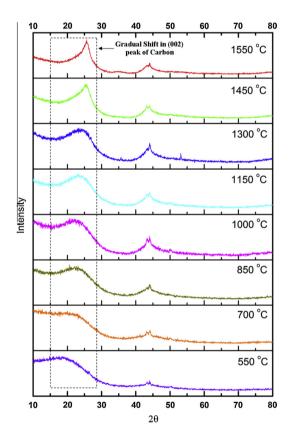


Fig. 3. Comparison of XRD patterns of chars prepared at different pyrolysis temperature.

3.2.3. FTIR analysis

FTIR analysis is a useful method for comparing the intensity of interested peaks in vibrational absorption spectra. The FTIR spectra of chars prepared by waste CDs at different pyrolytic temperature are shown in Fig. 7. Each spectrum is a spectral average of at least 10 scans. The peaks due to carbon dioxide and water vapour have been subtracted, and increasingly upward drift in the baseline towards higher wave numbers were also corrected in order to get quality spectra. The drift seen at higher temperature chars was probably due to increase in carbonised content [21]. Infrared band assignment for waste CD indicates that it contains C—H group, C=O group, C—O group, aromatic groups by its overtones and oop bands (Fig. 6). As seen from Fig. 7, the spectra of char pyrolyzed at 550 °C looks similar to waste CD but the intensity of all

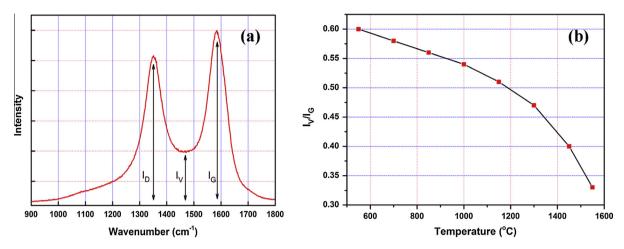


Fig. 4. (a) Raman spectra of char from pyrolysis at 1550 °C and (b) I_V/I_G ratio of chars obtained at different rapid pyrolysis temperatures.

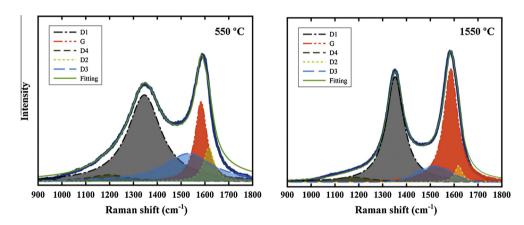


Fig. 5. Curve fitting of Raman spectra of chars obtained at 550 $^{\circ}\text{C}$ and 1550 $^{\circ}\text{C}$ temperatures.

peaks were decreased significantly. The reduced intensity of peaks in spectra clearly indicates that chemical structure of waste CD has changed at temperature 550 °C, the contents of C=O and C-O groups were almost invisible, which were high intensity peaks in waste CD. Although C=O and C-O groups are less thermally stable above 550 °C when they are compared to aromatic and methyl structures, but still they were found to be visible at this temperature. In spectra of char at 700 °C, the C=O and C-O peaks disappeared, and the intensity of methyl and aromatic peaks was drastically decreased. This clearly indicates that at 700 °C, the carbonate groups were completely degraded and aromatic groups were also hardly visible. At pyrolysis temperature 850 °C, the chars were devoid of peaks in aromatic overtone and 'oop' region, which indicates the absence of any functional groups like carbonate or aromatic structures in char. At 1000 °C or above pyrolysis temperature, the spectra of chars were devoid of all peaks and seemed to be similar to coke. The increase in upward drift (corrected in spectra) with increase in pyrolysis temperature clearly indicates an increase in aromatic contents.

3.2.4. XPS analysis

Fig. 8 shows the convoluted C1s spectra fitted using linear background for waste CD char obtained at rapid pyrolysis temperature 550 °C and 1550 °C. The calculated results for C1s peak such as start binding energy (BE), peak BE, end BE, full width half maximum (FWHM) and atomic compositions (at.%) for 550 °C and 1550 °C chars are shown in Table 3. For 550 °C waste CD char, the C1s core peak was separated into five components (C1s,

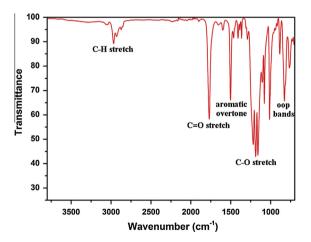


Fig. 6. FTIR spectrum of waste CD.

A1–E1), while at 1550 °C it was separated into six components (C1s, A2–F2) using Lorentzian curve fitting method. For char obtained at 550 °C, the major component was observed at 285.04 eV binding energy value (C1s, A1) with atomic concentration of 87.15%. The A1 component generally belongs to C—C and C—H bonds. The other components belong to various C=O and C—O groups as confirmed by FTIR also (see Fig. 7). For char obtained at 1550 °C, the atomic concentration of C—C and C—H bonds was found to have decreased to 22.31% (C1s, B2) and also

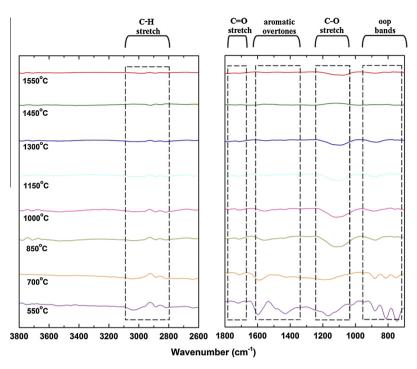


Fig. 7. FTIR spectra of chars obtained at different rapid pyrolysis temperatures.

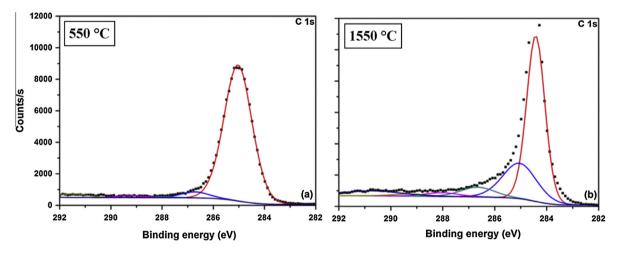


Fig. 8. XPS spectra of convoluted C1s fitted using linear background for chars obtained at (a) 550 °C and (b) 1550 °C temperatures.

Table 3XPS peak positions of fitted C1s at 550 °C and 1550 °C temperatures.

Name	Start BE	Peak BE	End BE	FWHM (eV)	Area (CPS eV)	At.%
PC char 550 °C						
C1s A ₁	294.38	285.04	281.38	1.28	12010.63	87.15
C1s B ₁	294.38	286.72	281.38	1.28	529.7	3.84
C1s C ₁	294.38	288.04	281.38	1.28	92.86	0.67
C1s D ₁	294.38	289.24	281.38	1.28	172.11	1.25
C1s E ₁	294.38	291.48	281.38	1.28	319.59	2.32
PC char 1550 °C						
C1s A ₂	293.88	284.42	281.28	0.83	9456.09	55.9
C1s B ₂	293.88	285.05	281.28	1.54	3773.83	22.31
C1s C ₂	293.88	286.65	281.28	1.54	1030.92	6.1
C1s D ₂	293.88	288.05	281.28	1.54	399.41	2.36
C1s E2	293.88	289.25	281.28	1.54	234.96	1.39
C1s F ₂	293.88	290.71	281.28	2.61	899.49	5.32

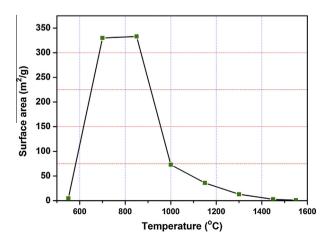


Fig. 9. Variation of BET surface area of chars obtained at different rapid pyrolysis temperature.

Table 4BET surface area of waste CD chars obtained at different pyrolysis temperature.

Temperature (°C)	BET surface area (m ² /g)		
550	4.6		
700	330.0		
850	334.2		
1000	73.1		
1150	36.0		
1300	13.2		
1450	2.8		
1550	0.9		

the highly ordered pyrolytic graphitic (HOPG) peak emerged at 284.42 eV (C1s, A2) and 290.71 eV (C1s, F2) binding energy values corresponding to —C=C— bonds [22,23]. The appearance of HOPG peak with atomic concentration 55.9% confirms the transformation of carbon to HOPG structure at rapid pyrolysis temperature

1550 °C. These results are in accordance with the XRD and Raman results (Figs. 3 and 5) of structural ordering of waste CD char as a function of temperature during rapid pyrolysis.

3.2.5. Surface area analysis

The BET surface area of chars obtained during rapid pyrolysis at different temperatures is presented in Fig. 9 and Table 4. The surface area of 4.6 m²/g was observed for 550 °C char, the low surface area is might be due to incomplete decomposition of waste CDs. But for 700 and 850 °C chars, high surface area was observed, may be attributed due to violent release of volatiles during decomposition of waste CD. The release of volatiles has favoured the formation of pores, which has led to the high surface area for chars obtained at 700 and 850 °C temperatures. The chars obtained after 850 °C showed gradual decrease in surface area with temperature and decreases to 0.8 m²/g for the char obtained at 1550 °C as seen from Fig. 9. At 1000 °C and above pyrolysis temperature, structural

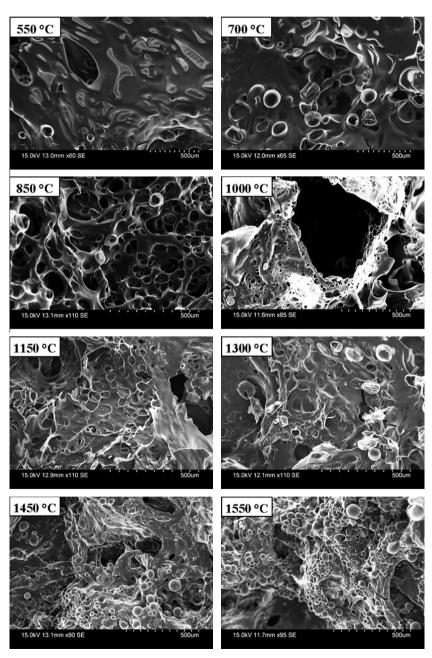


Fig. 10. SEM observation of chars obtained at different rapid pyrolysis temperatures.

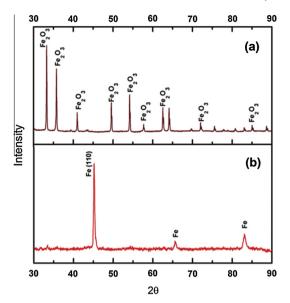


Fig. 11. XRD spectra of (a) hematite and (b) reduced sample at $1400\,^{\circ}\text{C}$ temperature.

ordering and merging of pores would decrease the surface area of chars. As a result of melting, fusing and carbonisation, the pores might be blocked and increases with temperature. The blockage of pores would prevent the adsorption gas to enter and hence low surface area was observed for chars obtained at and above $1000\,^{\circ}\mathrm{C}$ pyrolysis temperature.

3.2.6. SEM analysis

The SEM image of chars produced at different temperatures during rapid pyrolysis is shown in Fig. 10. The different surface morphology was observed for chars obtained at different temperatures. The surface morphology of char at 550 °C showed that decomposition has occurred; vesicles and the few pores were clearly observable. The vesicles were the result of volatile gases released during decomposition of waste CD. At higher pyrolysis temperature i.e. at 700 and 850 °C, the formation of pores was clearly visible form SEM images. The pores were formed due to violent release of volatiles during decomposition; hence SEM image of 850 °C shows the numerous pores. The BET surface area was also high (Fig. 9) for 850 °C char is due to existence of numerous pores. When the pyrolysis temperature increased to 1000 °C, the char shows the sign of softening and fusing, which resulted in the reduction of pores. As pyrolysis temperature increased, the pore

number reduces and softening increases, as a result surface area decreases. The ordering and structural arrangement in chars were clearly visible for 1000 °C char and increased significantly with higher pyrolysis temperature.

3.3. Reduction of Iron oxide

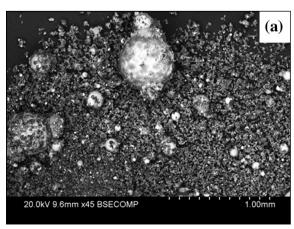
Otsuka and Kunii [24] found that the reduction of iron oxide to metallic iron was significantly enhanced when graphite was used as a reductant; this increase was attributed to the catalytic activity of metallic iron on the gasification of carbon. The graphitic structure of carbon plays an important role in enhancing the reduction and further enhancing the carburization of iron [25]. XRD, Raman and XPS results of pyrolyzed char has shown the transformation of carbon structure from disordered to highly ordered pyrolytic graphitic (HOPG) nature at high temperature. Hence for this study we have used char obtained at 1550 °C for reduction of iron oxide studies.

Fig. 11a shows the XRD pattern of haematite used for reduction studies. XRD pattern of reduced iron oxide at 1400 °C by using waste CD char as reductant is shown in Fig. 11b. The appearances of peaks corresponding to Iron (Fe) phases were observed in XRD pattern. The Fe phase peak (110) appeared to be dominant and absence of haematite phase clearly indicates complete reduction. The off-gases generated and degree of reduction as a function of time during the reduction of iron oxide at 1400 °C is shown in Fig. 12a. The cumulative moles for CO and CO_2 were 1.20×10^{-2} and 0.66×10^{-3} respectively at 1400 °C. The initial CO evolution started after 2 min of reaction and reached to maximum reduction after 4 min of reaction, and 90% reduction was achieved for 20 min of reaction time. A morphological feature of reduced sample was investigated by SEM. Fig. 12b shows the SEM images of reduced samples at 1400 °C and presence of metallic iron was clearly seen, and spherical shape of metallic iron appeared more bright and larger in size. This study evidently confirms that waste CDs char can be used as a promising supplementary carbon source in ironmaking.

4. Conclusions

In this study, the structural evolution of waste CD chars during rapid pyrolysis at different temperatures from 550 to 1550 °C has been investigated. The main conclusions are summarised below.

(a) The char yield decreased from 26% to 19% when pyrolysis temperature increased from 550 to 1550 °C. The increase in carbon/oxygen ratio was observed in chars indicating the increase of aromatic and carbonaceous nature.



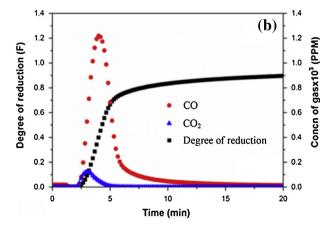


Fig. 12. (a) SEM morphology of reduced iron oxide samples and (b) Off-gases generated and degree of reduction as a function of time at 1400 °C during reduction of iron oxide.

- (b) As pyrolysis temperature increased, the char became progressively more aromatic and ordered. XRD, FTIR, XPS and Raman analysis confirms gradual decrease of carbonyl and aliphatic groups with higher pyrolysis temperature.
- (c) From SEM analysis, the development of pores in chars was clearly observed and increased from 550 to 850 °C pyrolysis temperature. At and after 1000 °C temperature, the pores decreased due to structural ordering and merging of pores.
- (d) Furthermore, the structurally ordered char obtained at 1550 °C was used as carbon reductant for iron oxide reduction process. The SEM, XRD and IR gas analysis confirms the reduction of iron oxide and 90% reduction was achieved. This result shows waste CDs as a promising supplementary carbon source in ironmaking industries and reduces the industry's dependence on conventional coal/coke.

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